DEVELOPMENT OF BATTERY SEPARATOR MATERIAL PROCESS

Mid-Program Report

by

L. M. Adams
W. W. Harlowe, Jr.
G. C. Lawrason

Project No. 01-2015

Contract No. 951718

Prepared for

Jet Propulsion Laboratory
California Institute of Technology
4800 Oak Grove Drive
Pasadena, California 91103

Attn: H. E. Patterson
Senior Contract Negotiator
Mail Station 190-212

February 15, 1967

SOUTHWEST RESEARCH INSTITUTE
SAN ANTONIO HOUSTON

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"This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, sponsored by the National Aeronautics and Space Administration under Contract NAS7-100."

February 15, 1967

Southwest Research Institute
8500 Culebra Road
San Antonio, Texas 78206

APPROVED:

John T. Goodwin Jr

John T. Goodwin
Director
Department of Chemistry
and Chemical Engineering

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I. SUMMARY

The optimization of the procedure for preparing a heat-sterilizable battery separator material from low-density polyethylene film has been initiated. It has been found that grafting of the film with acrylic acid with subsequent crosslinking with divinylbenzene yields a more uniform product having more desirable electrical properties than that produced when the film is crosslinked first and then grafted.

II. INTRODUCTION

A sterilizable battery separator material was developed by RAI for the Jet Propulsion Laboratory. This material was prepared by crosslinking low-density polyethylene film with divinylbenzene and subsequently grafting the resultant film with acrylic acid. Radiation from a cobalt-60 source was used to initiate both reactions.

Attempts by other contractors to use the procedure developed by RAI were unsuccessful. In many cases, little or no grafting occurred, and retreatment of the film with acrylic acid was required to obtain appreciable grafting. The product in all cases was very nonuniform and, as a consequence, unsuitable as a battery separator material.

The purpose of this experimental program is to study the basic procedure for preparing the battery separator material and optimize the procedure so that film having uniformly low electrical resistance can be produced.

Initially, the study was to be directed toward the acrylic acid grafting procedure of the divinylbenzene-crosslinked polyethylene film. The parameters to be studied were irradiation dose rate, total irradiation dose, temperature during irradiation, film washing procedures, and the exclusion of oxygen from the grafting solution during irradiation.

The desired properties of the final film are a maximum electrical resistance of 0.120 ohm-inch^2 for an approximately 1-mil film and a minimum wet tensile strength of 700 psi.

III. EXPERIMENTAL PROGRAM

A. Test Plan

A test plan for the proposed work was submitted and approved. A copy of this plan is reproduced in Appendix A of this report. The test plan is based on a factorial experiment to study irradiation dose rate, total irradiation dose, continuous and discontinuous irradiation, temperature during irradiation, and the presence and absence of oxygen. Several preliminary experiments were to be made to determine the desirability of removing the inhibitor from the crosslinking and grafting solution.

B. Nomenclature

Throughout this work, 30-foot rolls of polyethylene film are modified, and to identify the various parts of the roll which are analyzed, the following procedures and nomenclature are used.

One-foot samples of film are taken from various parts of the rolls of modified polyethylene film for analysis. These samples are identified by roll number, position along the length of the roll, and position across the roll. The designation "10-1, middle" represents roll number 10, the first foot of the roll, and the center part of the sample respectively. As the nomenclature applies to the modified film, the position along the roll may be greater than 30, as the film increases in length as a result of the treatment.

At times, the rolls are split into pieces for further treatment. When this occurs, the outer and inner portions of the roll are identified by the superscripts O and I. As an example, "10^O" and "10^I" are the outer and inner portions respectively of roll 10.

When both crosslinking and grafting procedures are performed on the same roll of film, the innermost part of the roll from the first treatment becomes the outermost part for the second treatment during the rerolling operation. As the grafting operation is the one being studied, all sample nomenclature is based on the position of the film during the grafting operation.

C. Irradiation Configuration and Dosimetry

Irradiation of the polyethylene sheet was performed in the Southwest Research Institute's Radiation Effects Facility and in much the same manner that was used in the "pilot" program that preceded the present contract. However, in the interim between the two programs, 11,000 curies of high specific activity cobalt-60 were added to the facility, and a completely new setup was designed to take advantage of this increased capability.

The target polyethylene roll width (and the roll container height) was essentially unchanged, so the vertical semi-parabolic cobalt-60 holder used previously was retained. Additional cobalt-60 sources were attached on the center line of the holder and evenly spaced in groups of six from top to bottom.

Mapping the gamma flux in the target area (front) of the new source indicated workable isodose volumes of 25,000 R sufficient to contain eight turntables and target cylinders. This volume was almost semicircular and symmetrical to the center line of the source, with the center cylinders offset slightly farther from the source. The arrangement is shown in Figure 1.

Detailed dosimetry using the Bausch and Lomb cobalt glass chip technique was performed after the target cylinders and turntable rack were fabricated. The entire assembly was adjusted, in position or source strength, until it was determined that the incident dose rates were those specified within the error of the dosimetry system. Dosimeters were placed within the target cylinders (in air) at the top, center, and bottom of the volume to be occupied by the polyethylene rolls and near the inside surface of the cylinder. The dosimeters were wrapped in lead foil to shield them from low-energy "knockout". The cylinders were rotating in each case so that the measured dose rate is an average of the small difference in rate across the diameter of each container. Results are presented in Table 1.

D. Crosslinking of Film with Subsequent Grafting

Eight rolls of polyethylene film were crosslinked with uninhibited divinylbenzene at 25°C. One roll was shipped to the Sponsor for his evaluation, one roll was examined for changes in tensile strength, and the remaining rolls were saved for further treatment. As expected, the tensile strength of the crosslinked film (Table 2) was higher than that of the untreated film.

Descriptions of the crosslinking procedure and the method of removing the inhibitor from the divinylbenzene are given in Appendix B.

Three rolls of the divinylbenzene-crosslinked polyethylene film were grafted at 25°C with acrylic acid using the grafting procedure described in Appendix B. As in previous work, the grafting was not uniform throughout the roll (Table 3). The electrical resistance of most of the film was excessively high.

Roll Number 2 was grafted with uninhibited acrylic acid (inhibitor removed by distillation of acid), while the others were grafted with inhibited acid. There appears to be a slight increase in the amount of grafting of Roll Number 2 as indicated by the lowering of the electrical resistance. However, a definite conclusion cannot be made as to the value of removing the inhibitor because of the nonuniformity of the grafting.

The amount of acrylic acid homopolymer was considerably less than obtained during the previous program, and the film could be removed readily from the jars. It is believed that the decrease in homopolymer was due to the removal of air pockets from the roll of film before irradiation.

All grafting solutions were monitored for temperature change during irradiation, and no exotherm occurred.

In a discussion of the above results with the technical representative of the Sponsor, it was agreed that an evaluation of polyethylene film which was grafted first and then crosslinked should be made before proceeding with the proposed test plan. It was felt that the crosslinking was interfering with the grafting reaction by limiting diffusion of the acrylic acid into the film and/or residual divinylbenzene or its decomposition products had an adverse effect on the grafting reactions.

E. Grafting of Film with Subsequent Crosslinking

Two rolls of untreated polyethylene film were grafted with acrylic acid. One roll (No. 10) was analyzed, and the results are tabulated in Table 4. Nonuniform grafting took place on the outermost four feet of the roll of film. This was determined by infrared studies, appearance and electrical resistance values.

The infrared instrument was set for 1540 cm^{-1} (6.4μ), and a strip of the film was moved through the film holder. No grafting was indicated on the first foot of film. There was intermittent grafting on the second, third, and fourth foot and continuous grafting after the fourth foot. When infinite absorbance was obtained, it was noted that the film had swelled considerably and had a reticulated appearance. When the absorbance was nil, the film had a smooth appearance much the same as the original untreated film. From four feet to the innermost part of the roll, the electrical resistance was well below the desired maximum value (Table 4). There was also considerably more swelling of this film during grafting than was obtained with the film that was crosslinked before grafting.

The amount of acrylic acid homopolymer formed during grafting was small, and the rolls of film could be readily removed from the jars.

One sample of the grafted film was sterilized for 72 hours at 142°C. The results (Table 5) were encouraging, as the film appeared to withstand the sterilization, and the electrical resistance remained well below the desired maximum value. The tensile strength met the minimum requirement.

Each of the aforementioned rolls of grafted film were divided into two equal parts. One part of each roll was crosslinked with inhibited divinylbenzene, and the other portion of each roll was crosslinked with uninhibited divinylbenzene. Properties of the products are listed in Table 6.

It is difficult to draw definite conclusions as to the effect of the inhibitor in the divinylbenzene because of the small number of analyses. However, the crosslinking appears to increase the tensile strength of the film without having a detrimental effect on the electrical resistance.

IV. CONCLUSIONS

Grafting of low-density polyethylene film with acrylic acid followed by crosslinking with divinylbenzene gives a product superior to that produced by reversing the grafting and crosslinking procedures. The grafting is much more uniform, and the electrical resistance is well below the desired maximum value.

Because of the above results, the program should be reviewed and a new test plan submitted for optimizing the preparative procedures.

FIGURE 1. IRRADIATION CONFIGURATION

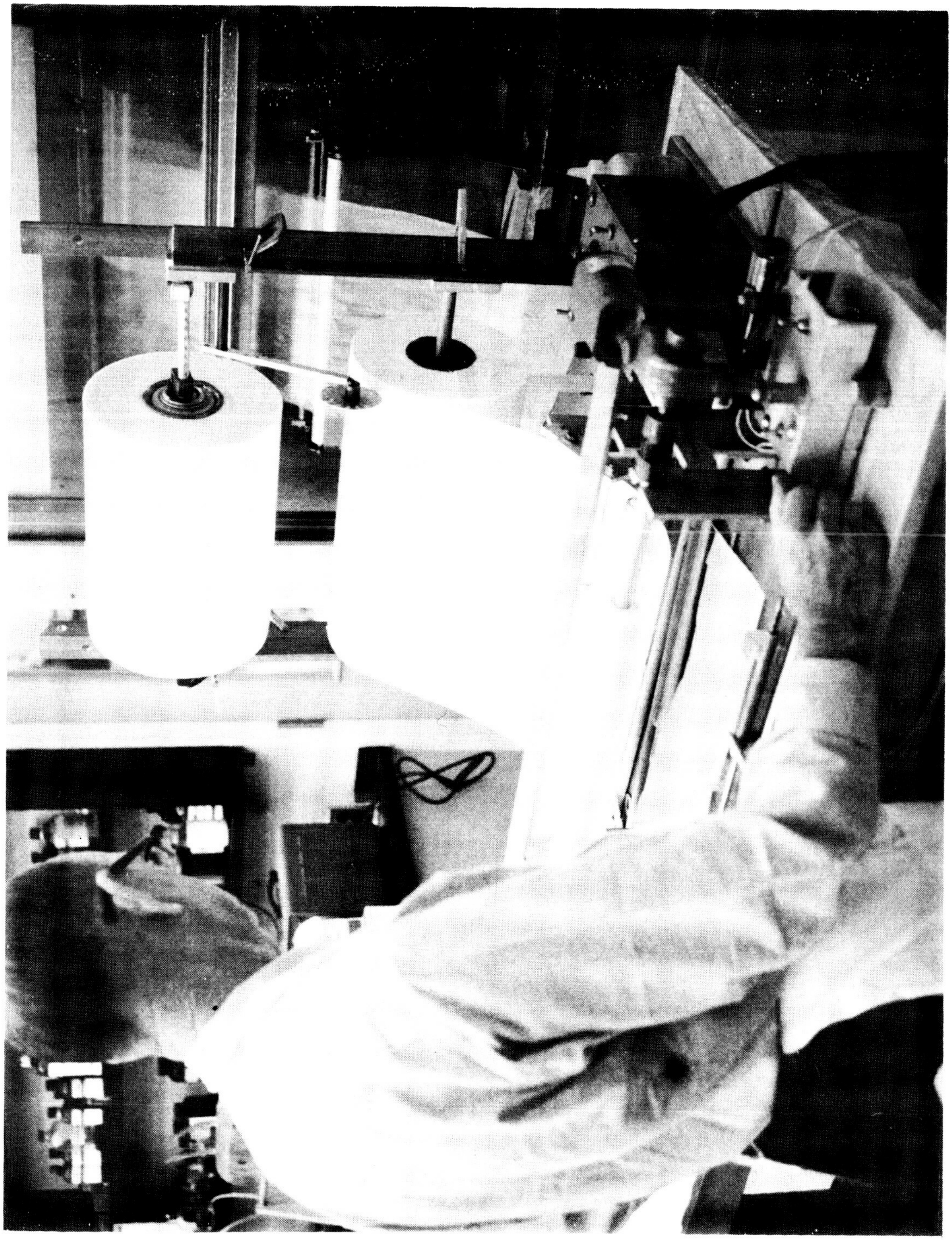

FIGURE 2. FILM-ROLLING EQUIPMENT

TABLE 1. DOSIMETRY

First Mapping at 34.5 in. base circle (1/2 arc)

Position		Dose Rate		
A	Top	4.4 x 10^4 R/hr		
	Center	4.6	"	"
	Bottom	4.1	"	"
B	Top	4.6	"	"
	Center	4.1	"	"
	Bottom	3.8	"	"
C	Top	3.8	"	"
	Center	3.8	"	"
	Bottom	3.6	"	"
D	Top	3.8	"	"
	Center	3.6	"	"
	Bottom	3.6	"	"

Continuation Table 1.

Second Mapping at 38.5 in. base circle (1/2 arc)

Position		Dose Rate		
A	Top	2.5 x 10^4	R/hr.	
	Center	2.7	"	"
	Bottom	2.6	"	"
B	Top	2.7	"	"
	Center	2.7	"	"
	Bottom	2.5	"	"
C	Top	2.4	"	"
	Center	2.5	"	"
	Bottom	2.5	"	"
D	Top	2.5	"	"
	Center	2.4	"	"
	Bottom	2.5	"	"

Third Mapping at 38.5 in. with A&B at 39.5 in. (1/2 arc)

Position		Dose Rate		
A	Center	2.4 x 10^4	R/hr.	
B	"	2.5	"	"
C	"	2.5	"	"
D	"	2.4	"	"

Continuation Table 1.

First Target Trial at 38.5 in. (full arc)

Position		Dose Rate
1	Top	2.15×10^4 R/hr.
	Center	2.15 " "
	Bottom	2.15 " "
2	Top	2.15 " "
	Center	2.40 " "
	Bottom	2.30 " "
3	Top	2.15 " "
	Center	2.30 " "
	Bottom	2.15 " "
4	Top	2.15 " "
	Center	2.15 " "
	Bottom	2.15 " "
5	Top	2.15 " "
	Center	2.30 " "
	Bottom	2.30 " "
6	Top	2.30 " "
	Center	2.30 " "
	Bottom	2.30 " "

Continuation Table 1.

First Target Trial at 38.5 in. (full arc) - Cont.

Position		Dose Rate
7	Top	2.30×10^4 R/hr.
	Center	2.30 " "
	Bottom	2.30 " "
8	Top	2.30 " "
	Center	--
	Bottom	2.30 " "

Second Target Trial at 38.5 in. (full arc)

Position		Dose Rate
1	Center	2.7×10^4 R/hr.
2	"	3.0 " "
3	"	2.7 " "
4	"	2.7 " "
5	"	2.6 " "
6	"	2.5 " "
7	"	2.5 " "
8	"	2.5 " "

Continuation Table 1.

Third Target Trial at 41.0 in. (full arc)

Position		Dose Rate
1	Top	2.0×10^4 R/hr
	Center	2.0 " "
	Bottom	2.0 " "
2	Top	2.0 " "
	Center	2.3 " "
	Bottom	2.4 " "
3	Top	2.1 " "
	Center	2.0 " "
	Bottom	2.0 " "
4	Top	2.0 " "
	Center	2.0 " "
	Bottom	2.1 " "
5	Top	2.0 " "
	Center	2.0 " "
	Bottom	2.0 " "
6	Top	2.0 " "
	Center	2.0 " "
	Bottom	2.1 " "

Continuation Table 1.

Third Target Trial at 41.0 in. (full arc) - Cont.

Position		Dose Rate
7	Top	2.1×10^4 R/hr
	Center	2.1 " "
	Bottom	2.1 " "
8	Top	2.0 " "
	Center	2.1 " "
	Bottom	2.0 " "

Fourth Target Trial at 41.0 in. (full arc)

Position		Dose Rate
1	Center	2.1×10^4 R/hr
2	"	2.1 " "
3	"	2.1 " "
4	"	2.1 " "
5	"	2.1 " "
6	"	2.1 " "
7	"	2.2 " "
8	"	2.1 " "

TABLE 2. TENSILE STRENGTH AND ELONGATION OF CROSSLINKED LOW DENSITY POLYETHYLENE FILM

Sample	Tensile Strength*, psi	Elongation, %
Control	1850	>100
(untreated film)	1735	>100
5-1, Middle	2410	>100
	2190	>100
5-12, Middle	2320	>100
	2370	>100
5-23, Middle	2320	>100
	1945	>100
5-30, Middle	2200	>100
	2320	>100

* Determined with a Gardner Film Tester.

TABLE 3. PROPERTIES OF POLYETHYLENE FILM WHICH WAS CROSSLINKED AND THEN GRAFTED

Sample	Tensile Strength, psi	Elongation, %	Thickness, mils	Resistance at 25.4 °C, milliohm-inch^2
JPL Control	550	19	1.2	25
(RAI-116)	1435	85	1.3	22
2-1, Top*	2370	>100	1.0	>3000
	2370	>100	1.0	>3000
2-12, Middle*	1945	>100	1.3	62
	1820	>100	1.3	139
2-23, Bottom*	1530	>100	1.4	245
	1350	99	1.4	57
2-32, Top*	1028	67	1.5	841
	-	-	1.5	>3000
3-1, Top	2730	>100	1.1	>3000
	-	-	1.1	>3000
3-6, Top	2120	>100	1.2	2500
	-	-	1.2	>3000
3-17, Middle	2120	>100	1.1	>3000
	-	-	1.1	>3000

* Inhibitor removed from acrylic acid prior to grafting.

TABLE 3. (Continued)

Sample	Tensile Strength, psi	Elongation, %	Thickness, mils	Resistance at 25.4 °C, milliohm-inch^2
3-30, Bottom	2020	>100	1.2	14
	-	-	1.2	214
4-1, Top	2210	>100	1.1	>3000
4-4, Top	-	-	1.1	1380
4-30, Top	-	-	1.1	>3000

TABLE 4. PROPERTIES OF POLYETHYLENE FILM WHICH HAD BEEN GRAFTED ONLY

Sample	Tensile Strength, psi	Elongation, %	Thickness, mils	Resistance at 25.4°C, milliohm-inch^2
10-1, Top	-	-	-	>3000
10-7, Top	1335	95	1.4	18
	840	85	1.5	15
10-13, Middle	1690	72	1.3	28
	1395	85	1.3	12
10-24, Bottom	1170	73	1.5	13
	1400	>100	1.5	2
10-35, Top	975	>100	1.4	20
	1060	>100	1.4	16
10-35, Middle	-	-	1.4	21
			1.4	11
10-35, Bottom	-	-	1.4	35
	-	-	1.4	16

TABLE 5. STERILIZATION OF GRAFTED POLYETHYLENE AT 142°C FOR 72 HOURS

Sample No. 10-7

Thickness, mils	
Before sterilization-dry	1.2
Before sterilization-wet	1.5
After sterilization-wet	1.6
Width, inches	
Before sterilization-dry	0.40
Before sterilization-wet	0.44
After sterilization-wet	0.46
Length, inches	
Before sterilization-dry	4.78
Before sterilization-wet	5.52
After sterilization-wet	5.44
Resistance at 25.4°C, milliohm-inch^2	
Before sterilization	17
After sterilization	9
Tensile strength, psi	
Before sterilization	1040
After sterilization	750
Elongation, %	
Before sterilization	90
After sterilization	53

TABLE 6. POLYETHYLENE FILM WHICH HAS BEEN GRAFTED AND THEN CROSSLINKED

Sample	Inhibitor in DVB	Tensile Strength, psi	Elongation, %	Thickness, mils	Resistance at 25.4 °C, milliohm-inch^2
9^{O}-1, Middle	no	2210	>100	1.1	>3000
		2120	>100	1.2	>3000
9^{O}-7, Top	no	-	-	1.1	3
9^{I}-17, Middle	yes	1490	>100	1.4	16
		1430	>100	1.4	13
10^{O}-8, Middle	yes	1285	>100	1.5	24
		1650	>100	1.5	18
10^{I}-14, Top	no	1610	>100	1.4	22
		1575	>100	1.5	24
10^{I}-23, Bottom	no	1620	>100	1.7	11
		1750	>100	1.6	11

APPENDIX A

Test Plan

SOUTHWEST RESEARCH INSTITUTE
8500 CULEBRA ROAD SAN ANTONIO, TEXAS 78206
Department of Chemistry
and Chemical Engineering

November 4, 1966

Mr. H. E. Patterson
Senior Contract Negotiator
California Institute of Technology
Jet Propulsion Laboratory
4800 Oak Grove Drive
Pasadena, California 91103

Re: SwRI Project 01-2015

Dear Mr. Patterson:

The following is a test plan for the development of a battery separator material process under Contract No. 951718 (Task Order Number RD-38 of Subcontract under NASA Contract NAS7-100).

The initial work will involve design of a radiation configuration, the setting up of suitable equipment for irradiation of multiple samples, and making necessary dosimetry measurements.

In this film modification process, low density (0.917) polyethylene film will be crosslinked with divinylbenzene under the influence of irradiation from a cobalt-60 source. The crosslinked film will be grafted with acrylic acid in a similar manner.

The crosslinking procedure will be that listed in the statement of work of Contract No. 951718. This procedure will not be modified unless later work indicates that this is necessary. Before making any modifications in this area, permission will be obtained from the Sponsor. A large quantity of crosslinked film will be prepared for use in the following studies.

The experimental portion of this program will be directed toward the optimization of the acrylic acid grafting procedure. The main parameters to be studied are:

(1) Irradiation dose rate

(2) Total irradiation dose

(3) Multiple irradiation in place of continuous irradiation to obtain total dose

(4) Temperature during irradiation

(5) Effect of oxygen

(6) Film washing methods (solution concentration, temperature, and time)

Before starting a systematic study of the above parameters, the following initial experiments will be made.

(1) Treat the polyethylene film or crosslinked polyethylene film with hot potassium hydroxide solution to remove caustic soluble materials. If the Sponsor obtains information on the light and heat

stabilizers (or other additives) present in the polyethylene film supplied, treatment will be modified for the removal of these additives.

(2) Remove inhibitor from the acrylic acid by distillation or liquid-solid chromatography to determine its effect on the grafting procedure.

If the above treatments indicate improvement of the acrylic acid grafted product, they will be utilized in subsequent work. Otherwise, the film and acrylic acid will be used as received.

In the above work it is planned to alternately evacuate and pressurize the test tubes containing the rolls of film so that as many as possible of the air pockets can be removed. The test tubes will remain open to the air during irradiation.

The following one-half replicate of a 2^5 factorial experiment will be followed for studying the parameters which are considered most important in the grafting of the crosslinked film with acrylic acid.

		High Dose Rate				Low Dose Rate			
		Continuous		Discontinuous		Continuous		Discontinuous	
		O_2	Inert	O_2	Inert	O_2	Inert	O_2	Inert
Total Dose 1	Temp 1	X			X		X	X	
	Temp 2		X	X		X			X
Total Dose 2	Temp 1		X	X		X			X
	Temp 2	X			X		X	X	

The following values will be used:

(1)	High dose rate	0.042 Mrads/hr
(2)	Low dose rate	0.021 Mrads/hr
(3)	Total dose 1	2.860 Mrads
(4)	Total dose 2	1.430 Mrads
(5)	T_1	ambient temperature
(6)	T_2	120°F
(7)	Discontinuous	on 16 hr, off 8 hr

After completion of this series of experiments, a test plan for further work will be submitted for your approval. The follow-up experiments will be determined by the results of the first series.

Washing of the grafted film should have no effect on the grafting reaction, although it may affect the properties of the final film. Because of this, washing studies will be delayed until the grafting experiments are completed, and the washing procedure used in the grafting studies will be that given in Contract No. 951718.

Properties to be determined on the grafted film are tensile strength, elongation, and electrical resistance. Samples for the determination of the above properties will be taken every 10 feet along the roll and at different places across the roll.

The radiation configuration design, equipment construction, and dosimetry will be started immediately. In this way, part of the preliminary work will be completed while negotiations on the test plan are being carried out.

Very truly yours,

Leon M. Adams

Leon M. Adams
Manager, Organic
and Polymer Section

APPENDIX B

Irradiation Techniques

CROSSLINKING PROCEDURE

1. A thirty-foot length of film is backed with absorbent crepe paper toweling and rolled onto a one-quarter inch aluminum pipe which is capped at one end.

2. The roll of film is placed in a hydrometer jar (75 mm x 550 mm) and covered with a one-percent (by volume) solution of divinylbenzene in a mixture of one part of benzene in 98 parts of methanol by volume.

3. The jar is alternately evacuated and allowed to return to atmospheric pressure until air bubbles can no longer be removed from the roll of film by evacuation.

4. The top of the jar is covered with aluminum foil to retard evaporation of the solution, and the film is allowed to equilibrate for at least 24 hours.

5. The jar and roll of film are exposed to a uniform cobalt-60 source adjusted to give a dose rate of 0.025 Mrads per hour until a total dose of 0.55 Mrads is obtained.

6. The jar is removed from the source and allowed to stand at least 24 hours. The film is unrolled and washed by passing it through benzene at a rate that permits a one-minute contact time with the benzene. The washed film is backed with paper toweling, rolled and permitted to dry.

INHIBITOR REMOVAL

1. The inhibitor was removed from the divinylbenzene, when required, by washing with approximately 9% potassium hydroxide solution. Four washings were made using a 4 to 1 volume ratio of divinylbenzene to potassium hydroxide solution. The divinylbenzene was then washed with distilled water until free of potassium hydroxide, dried by pouring through a column of white Drierite, and stored under nitrogen at -10°F until used.

GRAFTING PROCEDURE

1. A thirty-foot length of film is backed with absorbent crepe paper toweling and rolled onto a one-quarter inch aluminum pipe which is capped at one end.

2. The roll of film is placed in a hydrometer jar (75 mm x 550 mm) and covered with an acrylic acid solution having a composition of 25% acrylic acid, 70% benzene and 5% carbon tetrachloride by weight.

3. The jar is alternately evacuated and allowed to return to atmospheric pressure until air can no longer be removed from the roll of film by evacuation.

4. The top of the jar is covered with aluminum foil to retard evaporation of the solution, and the film is allowed to equilibrate for at least 24 hours.

5. The jar and roll of film are exposed to a uniform cobalt-60 source adjusted to give a dose rate of 0.021 Mrads per hour until a total dose of 1.430 Mrads is obtained.

6. The jar is removed from the source and allowed to stand at least 24 hours. The film is unrolled and washed in hot (80°C) aqueous 5% potassium hydroxide solution for one hour.

7. The film is then washed in hot (80°C) water for one hour and allowed to dry on paper toweling.